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VAPOR-PHASE INFRARED ABSORPTIVITY COEFFICIENT OF PINACOLYL METHYLPHOSPHONOFUORIDATE

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EXECUTIVE SUMMARY

We measured the vapor-phase absorptivity coefficient of pinacolyl methylphosphonofluoridate (GD, Soman) in the mid-infrared (IR). We used agent filled saturator cells suspended in a temperature controlled liquid bath to generate continuous streams of the compound diluted in nitrogen, which were sent to a variable path White cell and measured using a high resolution research grade Fourier transform IR spectrometer. The purity of the feedstock was verified by nuclear magnetic resonance (NMR) spectroscopy, gas chromatography (GC)-mass spectrometry, and GC-IR. The mass of GD in the vapor was determined with a gravimetric method, and the ratio was verified with thermal desorption GC. Ten spectra at different concentration-pathlength products were processed line by line through least squares analysis using MatLab® to produce the absorptivity coefficient of the compound and the statistical uncertainty in the data. Uncertainties in the data, expanded to a confidence interval of 2σ ($P = 0.95$), are Type-A: 2.7% and Type-B: 2.8% of the absorptivity coefficient. In this report, we compare our results to the data obtained by another laboratory using a different vapor generation method.

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PREFACE

The work described in this report was performed under the direction of the Detection Capability Officer, Defense Threat Reduction Agency Joint Science and Technology Office. This work was started and completed in September 2008.

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VAPOR-PHASE INFRARED ABSORPTIVITY COEFFICIENT OF PINACOLYL METHYLPHOSPHONOFUORIDATE

1. INTRODUCTION

We determined the high resolution vapor-phase absorptivity coefficients of the nerve agent, pinacolyl methylphosphonofluoridate (GD, Soman) in the spectral range of 4000-550 cm^{-1} in units of $(\mu\text{mol/mol})^{-1}\text{m}^{-1}$ and computed the uncertainties in the data. Previous efforts at the U.S. Army Edgewood Chemical Biological Center (ECBC) to obtain the absorptivity coefficients of this compound date back to the 1960s and were performed at lower resolution on a grating spectrometer.¹ Given the extensive use of vapor-phase infrared (IR) spectra for standoff detection of chemical warfare agents (CWAs), the need for current data is apparent.

Soman has the Chemical Abstracts Service (CAS) Registry Number 96-64-0 and is indexed under the name: phosphonofluoridic acid, methyl-, 1,2,2-trimethylpropyl. The molecular formula is $\text{C}_7\text{H}_{16}\text{FO}_2\text{P}$ and it has a molecular weight of 182.178. Other synonyms for the compound include 3,3-dimethyl-n-but-2-yl methylphosphonofluoridate; methyl pinacolyl phosphonofluoridate; pinacolyl methylfluorophosphonate; Zoman; and EA-1210. The structure is shown in Figure 1.

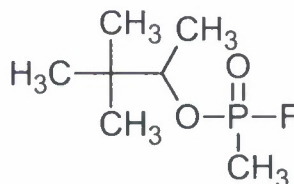


Figure 1. Structure of Pinacolyl Methylphosphonofluoridate (GD)

Pinacolyl methylphosphonofluoridate is a nerve agent that was first synthesized in Germany in 1944 although it was not used in combat during WWII. The vapor pressure of the compound at several temperatures is: 36.4 Pa (20 °C), 53.4 Pa (25 °C), and 101325 Pa (197.8 °C, calculated boiling temperature).²

2. EXPERIMENTAL PROCEDURE

2.1 Instrumental Details.

The system used to generate the continuous vapor stream was an adaptation of the saturator cell method developed at ECBC for measuring the volatility of CWA related compounds.³

The method, modified to generate continuous streams of chemical compounds for obtaining quantitative vapor-phase IR spectra, has been used to measure the absorptivity coefficients of benzene,⁴ as well as a variety of CWA related compounds.⁵ References 4 and 5

describe the experimental setup as well as the data collection and post-processing in more detail. The saturator passes a stream of nitrogen carrier gas, obtained from the boil-off of a bulk liquid nitrogen tank, across a cylindrical alumina wicking mechanism closed at one end in a glass holder filled with the analyte. This creates a saturated vapor-liquid equilibrium of the analyte on the downstream side of the saturator cell. The concentration of the analyte is determined by the temperature of the liquid phase. By suspending the saturator cell in a constant temperature bath, the concentration of the analyte can be predicted by its vapor pressure at the temperature of the bath. The Quantitative FTIR Laboratory is equipped with a Brooks (Brooks Instrument Co., Hatfield, PA) Model 5850S mass flow controller to maintain a constant flow to the saturator cell along with a second mass flow controller to add diluent to the stream, providing an additional means of adjusting the concentration of the compound delivered to the White cell of the FTIR. Linearity of the S series mass flow controllers is adjusted using a second order polynomial, resulting in uncertainties of approximately 1% or better of the flow rate at rates $\geq 25\%$ of full scale.

Spectra were obtained with a Bruker (Bruker Optics, Billerica, MA) Model IFS/66V Fourier transform IR spectrometer (FTIR). The instrument is equipped with both deuterated triglycine sulfide and mercury-cadmium-telluride (HgCdTe) detectors and is capable of obtaining spectra with a maximum spectral resolution of 0.1125 cm^{-1} (unapodized). The interferograms were recorded from $15798\text{--}0\text{ cm}^{-1}$ with a resolution of 0.125 cm^{-1} . Absorbance (log base-10) spectra were processed with boxcar apodization and 2X zero filled to obtain a data spacing of 0.0625 cm^{-1} . The instrument is equipped with a variable path White cell. The experimental data used path lengths of 2.727 and 5.377-m. The temperature of the White cell was maintained at $23 \pm 0.1\text{ }^{\circ}\text{C}$ by using a thermostatically controlled chamber enclosing the spectrometer and cell. Data were acquired at a speed of 60 KHz (HeNe laser zero crossing frequency) using the HgCdTe detector. Single beam spectra of the CWA were ratioed against spectra of clean, dry nitrogen. To minimize the effects of nonlinearity in the detector, the interferograms were processed using the proprietary Opus nonlinearity correction function. All interferograms were archived enabling further post-processing of data.

Temperature and pressure data were recorded using National Institute of Standards and Technology traceable digital manometers and thermometers, and all the data were archived. Concentration-pathlength products (CL) were computed in units of $\mu\text{mol/mol(m)}$ (ppm-m). A differential pressure manometer had previously been used to measure the dynamic pressure in the White cell with gas flowing into the cell, and the ambient pressure was plotted versus the differential pressure. The resulting equation was used to correct the readings from the ambient pressure manometer to the pressure in the White cell. The concentration-pathlength data were corrected to 296 K and $1.0132 \times 10^5\text{ Pa}$ (760 Torr) using the ideal gas law.

2.2 Feedstock.

The material used to generate the vapor streams for the experiments was purified by fractional distillation to remove the stabilizer. The purity was determined to be 101.6 ± 1.4 mol% ^{31}P nuclear magnetic resonance spectroscopy (NMR) using an internal standard; and 99.8% gas chromatography-mass spectrometry (GC-MS), with 0.2% pinacolyl alcohol. Analysis by GC-IR confirmed the presence of the pinacolyl alcohol in the feedstock. Sampling of the vapor effluent by thermal desorption-GC with a flame ionization detector indicated a purity of 99.0%. The spectra of the vapor from the saturator cell showed the presence of both water vapor and hydrogen fluoride in the effluent at a combined mass concentration that ranged from 0.25 to 0.5%. We used a concentration of 99.5 ± 1.0 (2 σ) mass% to compute the concentration-path length products of the vapor.

3. RESULTS AND DISCUSSION

Three trials were run to obtain spectra at 11 concentration-pathlength products. A trial is defined as filling and weighing the saturator cell, suspending it in the bath, applying a stream of nitrogen for a measured time, acquiring several spectra, stopping the nitrogen and removing it from the bath, and reweighing the saturator cell after drying the exterior surfaces and re-equilibrating to room temperature. The constant temperature bath was maintained at 23.0 °C for all three experiments.

Trace water vapor and hydrogen chloride were observed in several spectra. The concentrations of the compounds in the vapor, which decreased over time, were computed using their absorptivity coefficients and the features of the impurities were removed by spectral subtraction. Baseline corrections of the spectra were performed with a two point linear subtraction using a MatLab algorithm written in-house. The baseline correction in no case exceeded 0.001 $A[A = -\log_{10}(T)]$.

The composite spectrum (absorptivity coefficient) was computed using spectra with concentration-path length products ranging 50-416 $\mu\text{mol/mol(m)}$ (corrected to 296 K and 101325 Pa). As an initial check of the quality of the data, Beer's Law plots of two spectral lines (1212.5 and 720.6 cm^{-1}) were calculated using MatLab. These plots showed that, at least for these two spectral lines, the data appeared to be well fitted with no points lying outside the 95% confidence limits for a repeated set, a repeated single x , or the 95% confidence limits for a Grubbs Test for Outliers⁶ (Figures 2 and 3).

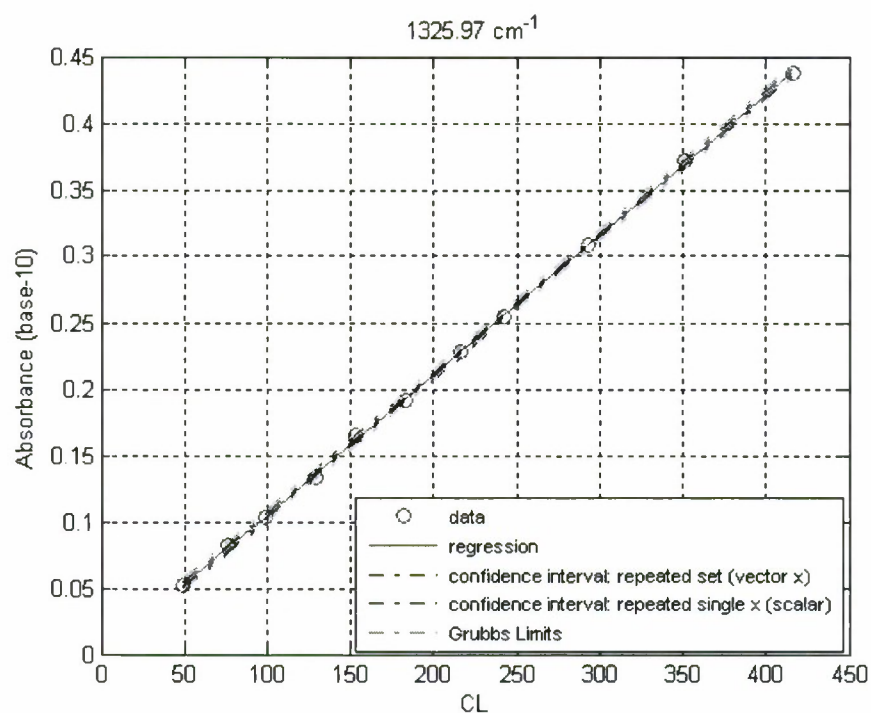


Figure 2. Beer's Law Plot of 1325.97 cm^{-1} Line in Vapor-Phase Spectrum of GD, Soman

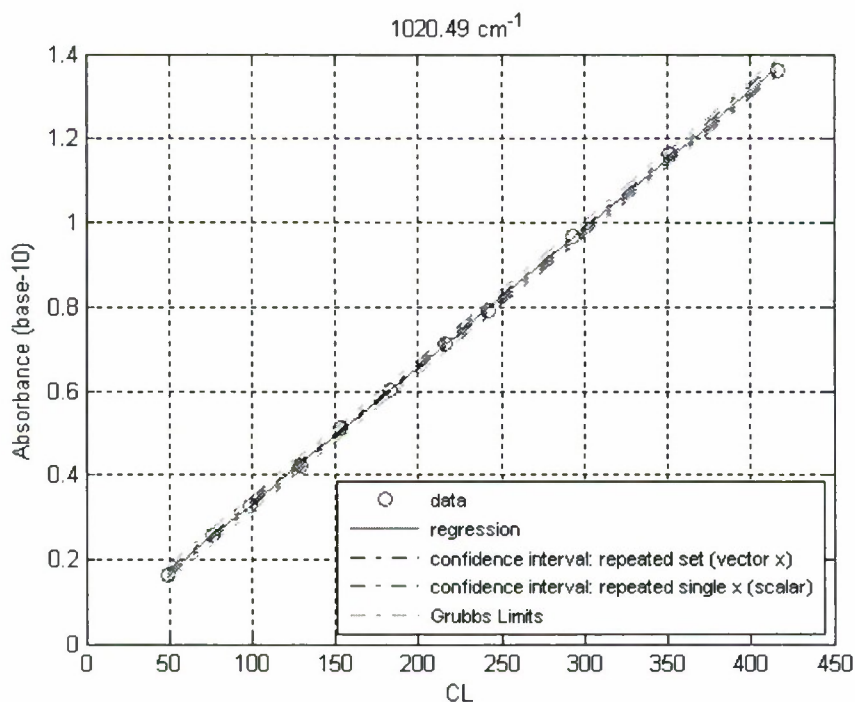


Figure 3. Beer's Law Plot of 1020.49 cm^{-1} Line in Vapor-Phase Spectrum of GD, Soman

The absorptivity coefficient (α) and uncertainty (Type-A, 2σ) were computed line by line within the spectral range of 4000-550 cm^{-1} using a MatLab program written in-house. Values of $(A = -\log T) > 1.5$ are normally assigned a weight of zero. Because all values recorded were for $A \leq 1.368$, all data were weighted at 1. Figure 4 is the plotted absorptivity coefficient (α), and Type-A uncertainty for the computed spectral range. The figures are plotted with α in $(\mu\text{mol/mol})^{-1}\text{m}^{-1}$. To obtain α in $(\text{mg/m}^2)^{-1}$, multiply the values in the mantissa of Figure 4 by 0.1326. This factor is derived from eq 1 using the molecular weight of Lewisite (182.178).

$$\frac{m^2}{\text{mg}} \left(\frac{24.15}{mw} \right) = \frac{\text{mol}}{\mu\text{mol}(m)} \quad (1)$$

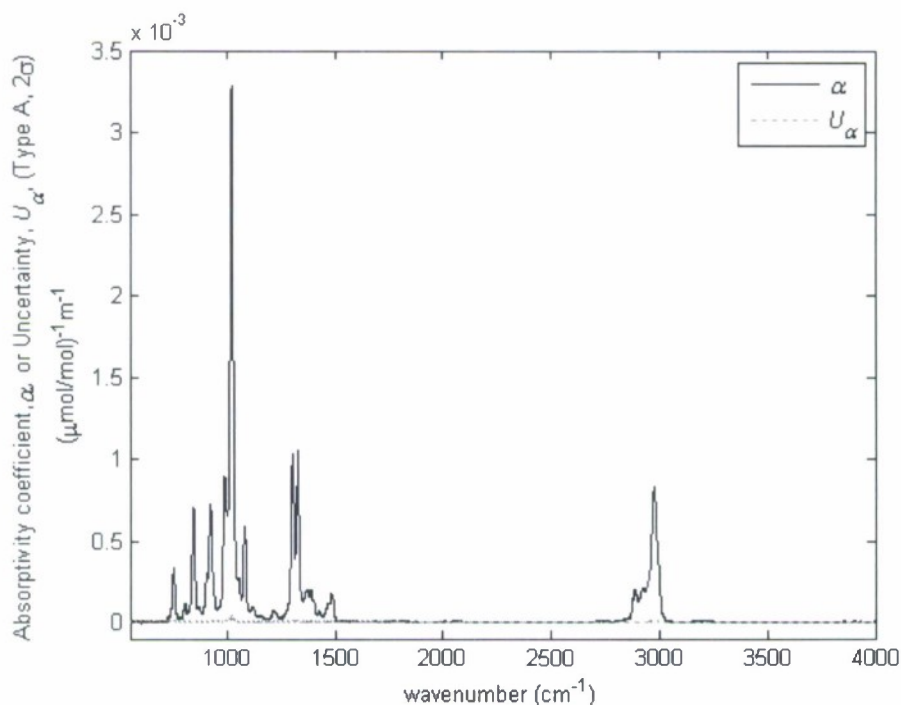


Figure 4. Absorptivity Coefficient and Statistical Uncertainty (Type-A, 2σ) for GD, Soman

Table 1 provides the absorptivity coefficients in $(\mu\text{mol/mol})^{-1}\text{m}^{-1}$ and $(\text{mg/m}^2)^{-1}$ for selected bands in units of wavenumber and micrometers (μm).

Table 1. Absorptivity Coefficient of Pinacolyl Methylphosphonofluoridate for Selected Bands

$\tilde{\nu}/(\text{cm}^{-1})$	2975.29	1325.95	1020.47	921.69
$\lambda/\mu\text{m}$	3.361	7.542	9.799	10.850
$(\mu\text{mol/mol})^{-1}\text{m}^{-1}$	8.314×10^{-04}	1.054×10^{-03}	3.274×10^{-03}	7.205×10^{-04}
m^2/mg	1.102×10^{-04}	1.397×10^{-04}	4.340×10^{-04}	9.551×10^{-05}

In general, expanded Type-A uncertainties were 1-1.5% of the absorptivity coefficient as seen in Figures 5 and 6. Figure 5 is a plot of absorptivity coefficients (abscissa) and fractional uncertainty (Type-A, U_A , 2σ) (mantissa).

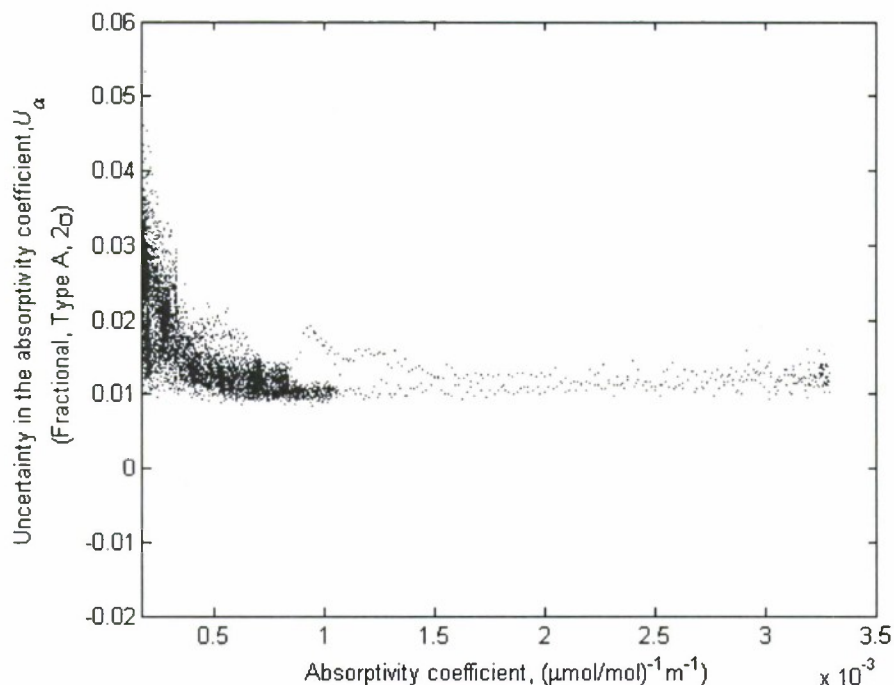


Figure 5. Absorption Coefficient (abscissa) and Type-A Uncertainty (Fractional, 2σ) for Soman

Figure 6 is a plot of the absorptivity coefficient and uncertainty. It includes a best fit of the data points obtained by least squares, which is an approximation of $U_A \approx ax+b$. For the fitted line in Figure 6, the coefficients are $A = 1.24 \times 10^{-2}$ and $b = 1.95 \times 10^{-6}$ (Table 2).

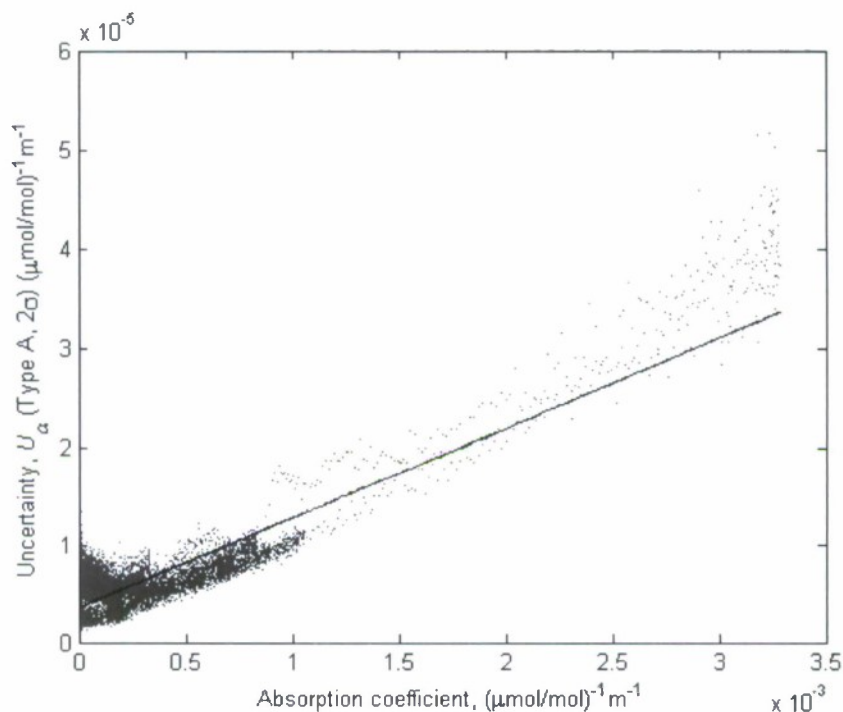


Figure 6. Absorption Coefficient (abscissa) and Type A Uncertainty (2σ) for GD, Soman. The line indicates the “best fit” obtained by least squares.

Table 2. Type-A Statistical Uncertainty for GD, Soman Vapor-Phase Absorptivity Coefficient

Type-A Uncertainty $2\sigma \approx ma + b$	
Slope m	Intercept b
1.24×10^{-2}	1.95×10^{-6}

Type-B estimated standard errors, along with their sources, as well as the combined Type-A and B uncertainties are provided in Table 3. The expanded combined Type-B uncertainty was computed using eq 2:

$$\Delta_B = (\Delta L^2 + \Delta T^2 + \Delta P^2 + \Delta FTIR^2 + \Delta NL^2 + \Delta MR^2)^{1/2} \quad (2)$$

The sources of uncertainty and their fractional values, as well as an explanation of the symbols in eq 1 are given in Table 4. The Type-A uncertainty of 2.7% (2σ) indicated in Table 3 is valid for values of the absorptivity coefficient $\geq 0.000133 \text{ (}\mu\text{mol/mol)}^{-1} \text{ m}^{-1}$ ($\alpha \geq 5\%$ of the peak of maximum intensity). For the most intense features, the Type-A uncertainty averages 1.2% of the absorptivity coefficient. Among the Type B uncertainties, the detector nonlinearity dominates at 0.01 (1σ).

Table 3. Uncertainties in Absorptivity Coefficient of GD, Soman from ECBC Data where $\alpha \geq 0.000164 \text{ (}\mu\text{mol/mol)}^{-1}\text{m}^{-1}$

Symbol	Fractional Deviation	Source
ΔL	0.005	Pathlength
ΔT	0.0006	Temperature of White cell
ΔP	0.002	Pressure
ΔFTIR	0.0005	Drift in spectrometer
ΔNL	0.01	Nonlinearity in detector
ΔMR	0.005	Mass rate
ΔD	0.005	Dilution rate
Δpurity	0.005	Purity of feedstock
Δ_B	0.028	Combined type B (2σ)
Δ_A	0.027	Type-A deviation (2σ)

4. INTERLABORATORY COMPARISON

Comparison of data between laboratories, especially when obtained using different methods, can be useful in assessing the experimental methods and for validating the accuracy of the data. One other source for comparing the high resolution absorptivity coefficient of pinacolyl methylphosphonofluoridate to the results acquired at ECBC are the data obtained by Pacific Northwest National Laboratory (PNNL) at Dugway Proving Ground.⁷ The PNNL database includes a detailed discussion of experimental methods and statistical processing. Each absorptivity coefficient spectrum is provided with a metadata file listing physical data for the compound, temperature and pressure range, number of individual spectra included in the fit of the absorptivity coefficient, and Type-A and Type-B uncertainties.

As can be seen in Figure 7, the spectra from ECBC and PNNL are qualitatively similar. The metadata file associated with the PNNL spectrum indicates uncertainties of 2.0% (Type-A) and $\leq 10\%$ (Type-B). A comparison of the integrated absorptivity coefficients from the two laboratories shown in Table 4, however, shows that the differences between the spectra appear to be larger than their uncertainties. As seen in Figure 8, the baselines of the two spectra differ significantly. In general, across the spectral range, the baseline of the PNNL spectrum appears to be shifted on the lower frequency (“red”) side of absorption features. While this may in part arise from the third order polynomial baseline subtraction used to correct the PNNL spectrum, we believe this is more likely the effect of scattering from aerosols present in the vapor of the spectra to compute the composite spectrum. The strong band associated with P = O stretch appears in GD at 1301 cm^{-1} in the vapor-phase and 1287 cm^{-1} in the liquid. Thus the simplest method to detect the presence of liquid in the vapor is to subtract an aerosol-free spectrum from the PNNL spectrum and compare the residuals in that region. Indeed, when the ECBC spectrum is subtracted from the PNNL spectrum, the residual (Figure 9) does show the presence of absorption features apparently associated with liquid-phase material in the vicinity of 1300 cm^{-1} . The aerosols are likely caused by the syringe pump method used to generate the vapor used for the PNNL spectra. The apparent presence of the liquid in the PNNL spectrum,

combined with the effects of aerosol scattering effects on the baseline, renders a quantitative comparison of the vapor spectra from the two laboratories difficult at best.

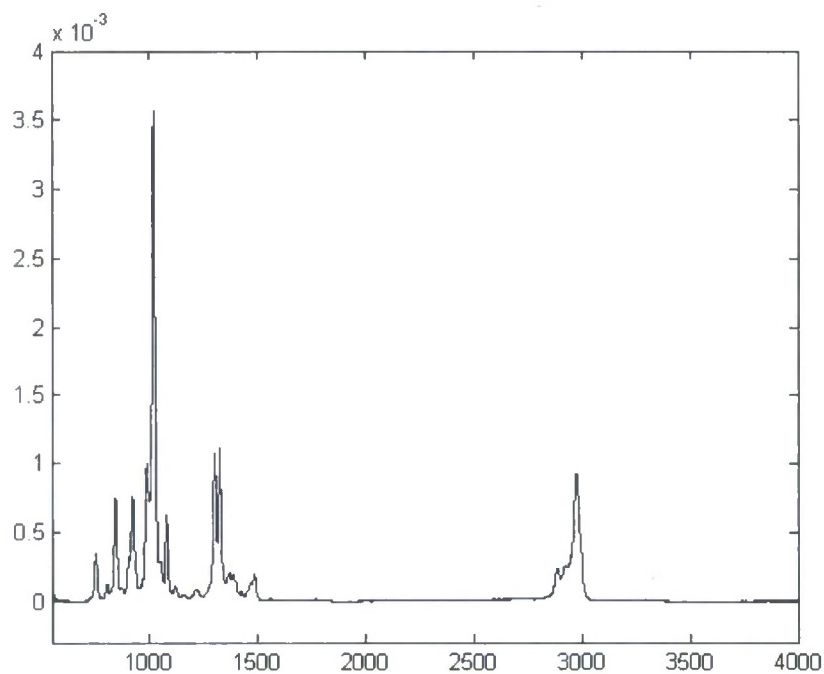


Figure 7. Absorptivity Coefficient of Vapor-Phase Soman from PNNL. The spectrum is qualitatively similar to the spectrum from our laboratory as seen in Figure 4.

Table 4. Comparison of Integrated Absorptivity Coefficients of GD, Soman from PNNL and ECBC

Range/cm ⁻¹	Integrated Absorptivity Coefficient		(ECBC-PNNL)/ECBC x 100
	PNNL	ECBC	
4000-550	0.2791	0.2476	-12.7%
1540-683	0.2131	0.1990	-7.1%
3258-2423	0.0593	0.0459	-29.2%

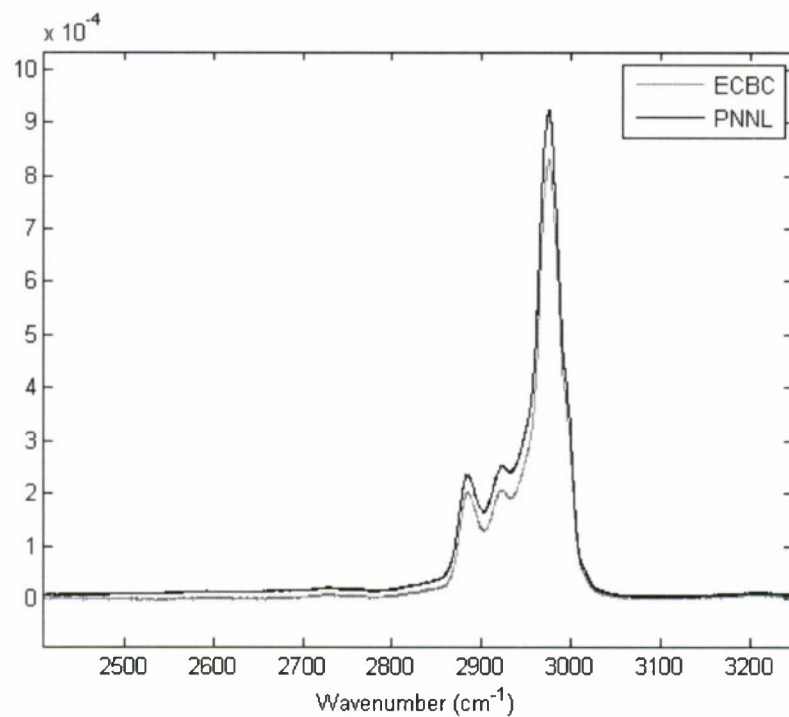


Figure 8. Absorptivity Coefficient Spectra in Vicinity of 3000 cm^{-1} from ECBC and PNNL. The PNNL spectrum appears to exhibit a baseline shift on the lower frequency side of the peaks associated with CH stretch. This may be indicative of aerosol scattering in the PNNL spectrum.

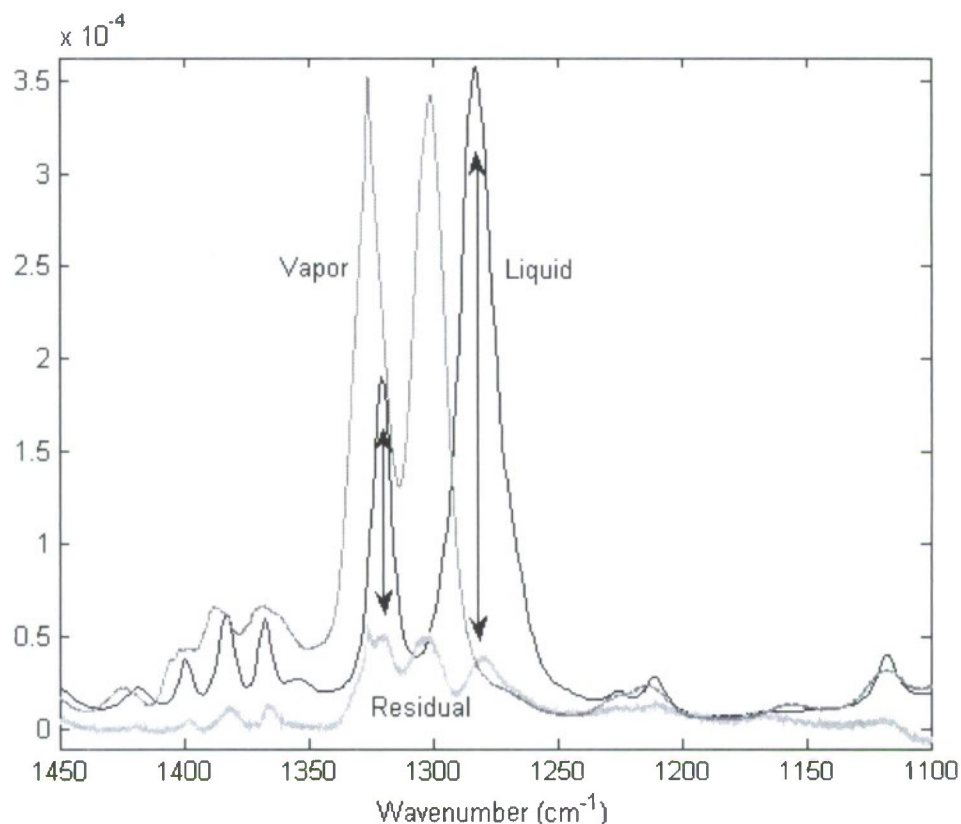


Figure 9. Absorptivity Coefficient Spectra of GD from ECBC (vapor) and PNNL (liquid) and Residual Spectrum Obtained by Subtracting PNNL Vapor Spectrum from ECBC Vapor Spectrum. Arrows indicate the presence of absorption features in the vicinity of 1300 cm^{-1} that are apparently associated with liquid-phase GD present in the vapor used to generate the PNNL vapor-phase spectrum.

5. CONCLUSIONS

We used a saturator cell system to acquire vapor-phase spectra of the nerve agent, pinacolyl methylphosphonofluoridate. These spectra were then used to compute the vapor-phase absorptivity coefficient at 0.125 cm^{-1} resolution. The uncertainties are Type-A = 2.7% and Type-B = 2.8% of the absorptivity coefficient for absorptivity coefficient $\geq 5\%$ of the peak of maximum intensity.

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